S GCAL ANALYTICAL LABORATION S. L.C.	SOP Number: HPLC-009	
	Revision: 00	
Extraction and LC/MS/MS analysis of Tributyl tetradecyl	Effective Date: 11/24/14	
phosphonium chloride (TTPC)		
	Supersedes Date: NA	

STANDARD OPERATING PROCEDURE FOR EXTRACTION AND HPLC/MS/MS ANALYSIS OF TRIBUTYL TETRADECYL PHOSPHONIUM CHLORIDE (TTPC)

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1.0 SCOPE AND APPLICATION

1.1 This procedure is designed for the analysis of Tributyl tetradecyl phosphonium chloride (TTPC) in a water matrix by dilution with acetone, filtration and analyzed by liquid chromatography/tandem mass spectrometry analysis.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water and Solid

3.0 SUMMARY OF TEST METHOD

- 3.1 A measured volume or weight of sample is transferred to a 40mL vial. Solvent is added to each vial and the sample/solvent is shaken for 1 minute. The samples are then filtered through a Whatman filter and analyzed by LC/MS/MS.
- 3.2 The target compound is identified by comparing the single reaction monitoring (SRM) transition and its confirmatory SRM transition if correlated to the known standard SRM transition.

4.0 **DEFINITIONS**

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

- 5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).
- 5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

- 6.1 Solvents, reagents, glassware and other sample extraction apparatus may yield interferences to sample analysis. All these must be demonstrated to be free from interferences under the conditions of analysis by analyzing method blanks.
- TTPC sticks to surfaces, if the glassware is not properly cleaned and rinsed with solvent, such as acetone, 2-propanol and acetonitrile, low ppt concentrations affecting the analysis may be found.
- 6.3 Automatic Pipettes with polypropylene tips are used with this method. The use of glass syringes for standards preparation, spiking, and calibration generate erratic results and should rinsed extremely well.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up

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procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable safety data sheets (SDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

- 7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.
- 7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.
- 7.4 Contact lenses are not to be worn in the laboratory.
- 7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

- 8.1 Extraction of Aqueous/Soil Samples
 - 8.1.1 VOA vials 20mL and 40mL
 - 8.1.2 2mL autosampler vials with Teflon lined crimp caps
 - 8.1.3 Gas Ultra pure Nitrogen
 - 8.1.4 Adjustable volume pipettes
 - 8.1.5 Pipettetips- various sizes of low retention coating
 - 8.1.6 Whatman syringe filter 0.2um (Catalog # 6750-2502)
 - 8.1.7 Plastic 10mL syringe with Luer Lock tip
 - 8.1.8 Analytical balance capable of weighing to 0.1g and calibrated daily when in use
 - 8.1.9 Gas-tight syringes 250uL, 500uL,1000 μL
 - 8.1.10 Volumetric flask, Class A- 10mL, 50mL, 100mL
 - 8.1.11 15 mL pipet, Class A

8.2 HPLC/MS/MS Analysis

- 8.2.1 HPLC Agilent Series 1260
 - 8.2.1.1 See SOP GEN-012 for Preventive Maintenance and Troubleshooting.
- 8.2.2 Column, Waters Acquity UPLC BEH C18, 2.1X100mm and 1.7um particle size, Waters part number 186002352
- 8.2.3 Agilent 6460 triple quadrupole mass spectrometer capable of MRM analysis
- 8.2.4 The Agilent system utilizes Agilent software MassHunter for instrument control and quantitation of all data.
- 8.2.5 The data system must be capable of time stamping all data produced with the correct date and time.

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9.0 REAGENTS AND STANDARDS

- 9.1 Reagents all organic reagent and solvent grades must be of Pesticide quality and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.
 - 9.1.1 Deionized water
 - 9.1.2 Acetone
 - 9.1.3 Methylene chloride
 - 9.1.4 Acetonitrile, HPLC grade
 - 9.1.5 Water, HPLC grade
 - 9.1.6 Isopropylalcohol
 - 9.1.7 Ammonium Acetate
 - 9.1.8 Sea Sand
- 9.2 Standards all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.
 - 9.2.1 (Tri-n-butyl)-n-tetradecylphosphonium chloride,(TTPC) 97%
 - 9.2.2 Stock Standard (TTPC, 10,000 μg/mL) weigh 0.1 g of TTPC standard into a 10 mL volumetric flask and bring to volume with 75% acetone/25% HPLC water. Store refrigerated at ≤ 6 °C in glass bottle with Teflon lined cap until use. Expires according to the manufacturer's expiration date or 1 year after receipt whichever is sooner or if comparisons with quality control check samples indicate a problem.
 - 9.2.3 TTPC Standard ($10 \mu g/mL$) add $100 \mu L$ of stock standard to a 100 mL volumetric flask and bring to volume with 75% acetone/25%HPLC water. Record in the Standards Preparation Logbook. Transfer into a glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months

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from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at \leq 6 °C until use.

- 9.2.4 TTPC (100 $\mu g/L$) add 1000 μL of TTPC 10 ug/mL standard to a 100 mL volumetric flask and bring to volume with 75%acetone/25% HPLC water. Record in the Standards Preparation Logbook. Transfer into a glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at \leq 6 °C until use.
- 9.2.5 TTPC Standard (1000 ng/L) add 500 µL of TTPC 100ug/L standard to a 50 mL volumetric flask and bring to volume with 75% acetone/25% HPLC water. Record in the Standards Preparation Logbook. Transfer into a glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.
- 9.2.6 Calibration Standards The calibration standards are prepared by diluting the TTPC 1000 ng/L standard into 6 working levels. Calibration levels: 25.0 ng/L, 50.0 ng/L, 100 ng/L, 250 ng/L, 500 ng/L, 1000 ng/L. The standards are diluted with 75% acetone/25% HPLC water.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

- 10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.
- 10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.
- 10.3 Samples are stored at the laboratory using conditions required by 40CFR 136 or reference method.
- 10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

- 11.1 Required preservation Cool to 0-6° C, but not frozen
- 11.2 Maximum holding time As per the EPA document CRL SOP # OM16 Rev0 the holding time has not yet been established for this analyte in the various matricies.

12.0 QUALITY CONTROL

- One method blank, LCS, LCSD, RLC, MS, and MSD shall be prepared per batch, a maximum of twenty samples.
- 12.2 If insufficient sample is available for a MS and/or MSD then document this on the Extraction Preparation Form.

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12.3 QC samples are prepared in the exact manner of the samples using the Procedure in Section 14.0.

13.0 CALIBRATION AND STANDARDIZATION

- Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.
- Auto-Pipettes will be calibrated each quarter and verified each day of use.

 Quarterly calibrations of auto-pipettes are recorded in the Non-Standard Labware
 Logbook and daily calibration verifications for auto-pipettes are recorded in the
 Daily Mechanical Pipet Calibration logbook associated with each pipette.
- 13.3 Retention Time Windows
 - 13.3.1 Retention time windows must be determined during initial instrument setup, when a new column is installed, when operating conditions are modified, or when major maintenance on the instrument occurs.
 - 13.3.2 For all analytes and surrogates a retention time study is performed over a minimum of 72 hours. The standard deviation (SD) is calculated based on this study using at least three determinations, measured to 0.001 minutes. The width of the retention time window is three times the SD for each of the analytes. Alternatively, if the calculated SD is less than 0.01 minutes, a default window of ± 0.03 minutes shall be employed.
 - 13.3.3 The daily retention time window for the analyte is equal to the center of the retentionwindow in the first CCV of the day, \pm 3 times the standard deviation calculated from the retention time study or \pm 0.03 minutes when the default is employed. If an ICAL is analyzed in the batch, use the midlevel calibration standard to set the daily retention time window for the analytical batch.

13.4 Tune

- 13.4.1 The Agilent 6460 is tuned monthly or when mass shifts of more than 0.2 Dalton are noticed by the analyst. The tuning solution used is Agilent 6460 tuning solution. The tune procedure for the 6460 is automated and can be found in the MassHunter software.
- 13.5 Initial Calibration (ICAL) an ICAL is performed using a blank and 5-6 standards analyzed at the concentrations noted in Section 9.2.6. An initial calibration curve must be analyzed and evaluated before any result can be quantitated. An initial calibration is performed daily.
- 13.6 Average Response Factor Calibration
 - 13.6.1 A minimum of five standards is required for this method. Calculate the average of the response factors and the standard deviation across the response factors. The lowest standard used must be at or below the reporting limit for each analyte. Use the average RF and the standard deviation to calculate the percent relative standard deviation (%RSD).

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When the response factors of the standards demonstrate less than 20%RSD for all target analytes, linearity through the origin can be assumed. If the RSD for any target analyte is greater than 20%, the analyst may wish to review the results for that analyte to ensure that the problem is not associated with just one of the initial calibration standards. Points may be dropped from the high and/or low end of the calibration curve if the minimum number of points and the project required detection limits are maintained. Points may not be removed from the middle of a calibration unless there is a documented reason. That point may be reanalyzed and replaced in the same analytical batch.

- 13.7 Linear Regression Calibration If the RSD exceeds 20%, a linear regression equation that is not forced through the origin may be used. The correlation coefficient must be at least 0.990 for the curve to be acceptable. The analyst should check the y-intercept (b). If the intercept is greater than half the reporting limit, this option should not be used.
- 13.8 Quadratic Curve Fit A quadratic curve fit may be used if the coefficient of determination $r2 \ge 0.990$. A minimum of a six-point calibration is used if this option is chosen and the curve shall not be forced through zero. The analyst should check the y-intercept (b). If the intercept is greater than half the reporting limit, this option should not be used.
- 13.9 Independent Calibration Verification (ICV) immediately following the ICAL analyze a standard containing all target analytes prepared from a solution that is independent from the ICAL standard. The ICV for TTPC is prepared from the same neat material as the calibration standard.
 - 13.9.1 The ICV is prepared at a concentration of 250 ng/L, and should not exceed a 30% difference from the initial calibration.
 - 13.9.2 If the criterion referenced above is not achieved, corrective action must be taken. This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation of the standards involved.
- 13.10 Continuing Calibration Verification (CCV) The CCV is analyzed at the end of the sequence or every 24 hours.
 - 13.10.1 The CCV is prepared and analyzed at a 250ng/L concentration, and must not exceed a 30% difference from the initial calibration.
 - 13.10.2 If the calibration verification standard fails to meet the criteria, repeat the injection of the standard. If the standard fails again, take appropriate corrective action (inspection of HPLC, re-prep standard, etc.). If the criteria cannot be met, a new calibration curve should be prepared.
 - 13.10.3 If the calibration verification standard analyzed after a group of samples has a response for an analyte that is high and the analyte was not detected in any of the previous samples during the analytical shift, then the samples do not need to be re-analyzed.

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14.0 PROCEDURE

14.1 Extraction of Aqueous Samples

- 14.1.1 Label each 40 mL VOA vial with the last six (6) digits of the sample ID to be extracted. Fill in the Extraction Preparation Form information as each item is completed.
- 14.1.2 With each extraction batch, a method blank, LCS, LCSD, and RLC are performed. The MS/MSD is performed when assigned.
- 14.1.3 With an Eppendorf pipette transfer 2.5 mL HPLC water into a 40 mL VOA vial labeled Blank; repeat for the LCS/LCSD and RLC. Record the volume on the Extraction Preparation Form. The RLC is spiked with 50 uL of a 5 ug/L TTPC standard.
- 14.1.4 Spike the LCS/LCSD and the MS/MSD with 50 uL of the 100ug/mL TTPC standard.
- 14.1.5 With an adjustable pipette transfer 2.5 mL of sample into a labeled 40 mL VOA vial. Record the volume on the Extraction Preparation Form.
- 14.1.6 To all QC and samples add 7.5 mL of acetone with an adjustable pipette and shake or vortex for 1 minute.
- 14.1.7 Filter the sample through the Whatman syringe filter by dispensing the first mL to waste and collecting the second mL in an auto sampler vial.
- 14.1.8 Sign the Extraction Preparation Form. Deliver a copy of the Extraction Preparation Form and the extracts to the HPLC-MS laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator with the Extraction Preparation Form.

14.2 Extraction of Soil Samples

- 14.2.1 Label each 40 mL VOA vial with the last six (6) digits of the sample ID to be extracted. Fill in the Extraction Preparation Form information as each item is completed.
- 14.2.2 With each extraction batch, a method blank, LCS, LCSD, and RLC are performed. The MS/MSD is performed when assigned.
- 14.2.3 Weigh 2.0g of sea sand into a 40 mL VOA vial labeled Blank; repeat for the LCS/LCSD and RLC. Record the volume on the Extraction Preparation Form. The RLC is spiked with 100 uL of a 5 ug/L TTPC standard.
- 14.2.4 Spike the LCS/LCSD and the MS/MSD with 50 uL of the 100ug/mL TTPC standard.
- 14.2.5 Weigh 2.0g of sample into a labeled 40 mL VOA vial. Record the volume on the Extraction Preparation Form.
- 14.2.6 To all QC and samples add 15 mL of acetone using a 15 mL pipet and shake or vortex for 1 minute.
- 14.2.7 Filter the sample through the Whatman syringe filter and collect in a 40 mL VOA vial.

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- 14.2.8 To all QC and samples add 5.0 mL of HPLC water using an adjustable pipet te.
- 14.2.9 Transfer approximately 1 mL into an autosampler vial.
- 14.2.10 Sign the Extraction Preparation Form. Deliver a copy of the Extraction Preparation Form and the extracts to the HPLC-MS laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator with the Extraction Preparation Form.

14.3 HPLC Conditions

- 14.3.1 Analytical column: Acquity UPLC BEH C18, 2.1x100mm, 1.7um particle size.
- 14.3.2 Injections of all standards and samples are normally made at a 40uL volume.
- 14.3.3 Standards and samples must be in 75:25 acetone :water.
- 14.3.4 Solvent A :20 mM ammonium acetate 95.25% acetonitrile, 4.75% HPLC water.
- 14.3.5 Solvent B: 20 mM ammonium acetate 95%HPLC water, 5% aceotnitile.
- 14.3.6 Ramp:

Time	Flow	SolventA	SolventB
0	0.3	0	100%
1	0.3	0	100%
4	0.4	100%	0
8	0.4	100%	0
9	0.4	0	100%
10	0.4	0	100%

14.4 Mass Spectrometer conditions

14.4.1 The instrument is set in the Electrospray(+) positive source setting.

14.4.1.1 Gas Temp: 350C 14.4.1.2 Gas Flow: 10L/min 14.4.1.3 Nebulizer: 35psi

14.4.1.4 Sheath Gas Temp: 380C 14.4.1.5 Sheath Gas Flow: 12L/min

14.4.2 Mass Spec parameters:

Compound	Primary/Confirmatory	SRM	Cone	Collision
		<u>Transition</u>	<u>(V)</u>	<u>(eV)</u>
TTPC	Primary	399.5-229.3	140	45
	First Confirmatory	399.5-75.9	140	46
	Second Confirmatory	399.5-343.5	140	40

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14.5 HPLC Analysis

- 14.5.1 Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with an initial calibration. It contains a CCV at the end of the sequence or 24 hours.
- 14.5.2 If the TTPC ions are present and the peak falls within the retention time window, then calculate the concentration present.
- 14.5.3 If a sample response exceeds the limits of the initial calibration of the system, a dilution of the sample must be analyzed which falls within the calibration range of the instrument. This should be done by diluting the sample with 75:25 acetone:water.
- 14.5.4 Manual Integrations See SOP QA-010 for GCAL's Manual Integration Policy.

15.0 CALCULATIONS

- 15.1 % Recovery (MS/MSD) = <u>Spike Concentration-Sample Concentration_x</u> 100 Spike Added
- 15.2 % Recovery (LCS/LCSD/Surrogate) = Spike Concentration x 100 Spike Added
- 15.3 RPD = $| \frac{\text{Concentration } 2\text{-Concentration } 1 | x 100}{(\text{Concentration } 2\text{-Concentration } 1)/2}$
- 15.4 Response Factor =A_s/C_s
 As= Peak Area of analyte
 Cs= Concentration of the analyte
- 15.5 % Difference = $[(RF_I RF_C) / RF_I] \times 100$ RF_I = Average response factor from initial calibration RF_C = Response factor from current verification check standard
- 15.6 % RSD = (SD/X) X 100

 RSD = Relative Standard Deviation

 X = mean of initial RF's for a compound

 SD = Standard Deviation of average RF's for a compound
- 15.7 Concentration, water samples $(ng/L) = \underline{[(Ax) (FV) (DF)]}$ [(RFX) (IV) (Vi)]

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Concentration, soil samples (ng/Kg) = [(Ax)(FV)(DF)][(RFX)(WS)(Vi)]

Ax = Area of compound being measured

FV = Final volume of extract

DF = Dilution factor

RFX = Response factor of compound being measured

IV = Volume of water extracted

WS = Weight of soil extracted

Vi = Volume of extract injected

15.8 Refer to SOP GEN-021 for calibration calculations.

16.0 REPORTING LIMIT/LIMIT OF QUANTITATION (LOQ)

- 16.1 Water samples -100 ng/L
- 16.2 Soil samples -250 ng/Kg

17.0 METHOD PERFORMANCE

17.1 Method performance is not available

18.0 POLLUTION PREVENTION

- Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.
- 18.2 Refer to OAM Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

- 19.1 The Method Blank concentration shall not be greater than half the Limit of Quantitation (LOQ) or 1/10 the amount measured in any sample (whichever is greater), or otherwise affect sample results. If the method blank fails to meet these criteria, the source of the contamination should be investigated and samples should be re-extracted and/or reanalyzed. If additional sample is not available, report with a case narrative and notify the GCAL project manager immediately.
- 19.2 The LCS control limits are used to determine batch acceptability. If the LCS fails to meet the laboratory acceptance criteria, the source of inaccuracy should be investigated and samples should be re-extracted and/or reanalyzed. Enter condition code "RP" for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately. If a target compound is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still

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- required to investigate the source of the failure. The LCS recovery limits for waters are 56-99% and solids are 70-130%.
- 19.3 The RLC (Reporting Limit Check Sample) is processed like a LCS just spiked near the reporting limit. This sample is to verify that if the analyte is present at the reporting limit it would be confidently identified. The RLC is performed with each extraction batch. The RLC recovery limits for water and solids are 70-130%.
- 19.4 The MS is evaluated against the LCS control limits and is used to determine sample matrix effect. If the MS recovery is outside of control limits and the LCS is within control limits, matrix interference is indicated. Report the results with a case narrative. The MS recovery limits for waters are 56-99% and soils are 70-130%.
- 19.5 The LCS/LCSD and MS/MSD RPD is evaluated using the criteria ≤ 30%. If the RPD is outside the control limits, investigate the source of the precision error. A source of precision error in the MS/MSD may be the heterogeneous nature of the sample. If a lab error is suspected, repeat the analysis. If a matrix issue is indicated, report the results with a case narrative.
- 19.6 Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before identifying a failure as matrix interference.
- 19.7 Native sample concentrations may be high in comparison to the spiking concentration and therefore an accurate recovery cannot be calculated. Document this in the case narrative.
- 19.8 Spikes may be diluted out in the analysis process if a 10X or higher dilution is performed. Document this in the case narrative.
- 19.9 Project specific criteria may apply. See SOP GEN-019.
- 19.10 For data review procedures and checklists, see SOP QA-002.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

- 20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:
 - 20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).
 - 20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 EPA – CRL SOP #OM16 Rev 0

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SOP for the analysis of TTPC in water/soil by MRM LC/MS/MS

23.0 METHOD MODIFICATIONS

Not applicable.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

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Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
07/17/14	00	New SOP	